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(72) Inventors WILLIAM SYDNEY DURHAM WISE,  
TERENCE GEOFFREY MARTIN and  
JOHN CHARLES WHITEHEAD

(19)



## (54) A METHOD OF DEGRADING RUBBER TO PRODUCE USEFUL MATERIALS

(71) We, COAL INDUSTRY (PATENTS) LIMITED, a company organised in accordance with the laws of Great Britain of Hobart House, Grosvenor Place, London, S.W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of degrading rubber and recovering useful material therefrom, particularly from vehicle tyres.

In view of the increased usage of motor vehicles, the problem of disposal of tyres is becoming serious. The rubber is not recoverable itself, and methods hitherto proposed for disposing of tyres have generally involved combustion. Complex equipment and high temperatures are generally required to ensure the complete combustion thereof, so that there is no pollution of the atmosphere by noxious fumes.

The present invention comprises a method of degrading rubber and recovering useful material therefrom, particularly rubber of the kind employed in vehicle tyres, comprising contacting the rubber with a solvent selected from water or organic solvents having a critical temperature of between 150°C and 450°C, preferably 250°C to 400°C, which solvent is in the gaseous phase, at above the critical temperature thereof, thereby degrading the rubber and dissolving at least part of the degradation products thereof in the gaseous phase, removing the gaseous phase from the solid residue of the rubber and condensing at least part of the degradation products of the rubber dissolved in the gaseous phase from the gaseous phase.

It is known in principle that gases at above, and preferably just above, the critical temperature thereof are particularly capable of dissolving into the gaseous phase liquid

or solid substances. The present invention lies in that, under these conditions, rubber, including vulcanised rubber, can be degraded. The volatile or volatilisable degradation products of the rubber are then dissolved into the gaseous phase. The non-volatile products particularly from vehicle tyres, comprise mainly the reinforcing carbon black which can be produced as a free-flowing powder.

The exact temperature at which any rubber will commence to degrade will depend on its structure. As a general guide, a temperature of the order of 250°C to 350°C is convenient but this invention is not limited to such a range. An important criterion will be that of the rate of degradation of the rubber. At too low a temperature, the rubber will not degrade. In general the range practical for most rubbers is likely to be 200°C to 400°C.

The choice of the solvent will in general depend on the temperature at which the method of this invention is to be carried out. It is preferred to carry out the method at just above the critical temperature and pressure of the solvent. It will generally be convenient not to employ a temperature of more than 100°C above the critical temperature of the organic solvent, and advantageously the temperature employed should not exceed 50°C above the critical temperature of the solvent. There may be other gases or vapours introduced with the organic solvent that are not above their critical temperatures. These may have the effect of promoting the degradative capacity and solvolysability of the organic solvent.

It is to be understood that certain solvents, particularly oxygenated solvents, may react with the rubber or degradation products thereof. Whether such solvents are suitable commercially can readily be found from an assessment of the mass balance and the value of the products. Compounds that

may be employed as solvents, alone or in mixtures, include aliphatic and aromatic hydrocarbons, aliphatic alcohols, ethers, ketones and esters, and halogenated (particularly chlorinated, fluorinated and chloro-fluorinated) derivatives thereof, as well as aliphatic and aromatic amines and heterocyclic compounds. Examples of solvents include pentane, hexane, octane, dodecane, cyclopentane, cyclohexane, benzene, toluene, ethylbenzene, xylenes, propanol, butanol, diisopropyl ether, anisole, ethyl acetate, butyl acetates, methyl propyl ketones, perfluoroacetone, carbon tetrachloride, chloroform, 1,2,2 - trichloro - 1,2,2 - trifluoroethane, n-propylamine, triethylamine, pyridine and dioxane.

The degradation products of the rubber may be condensed from the gaseous phase in which they are dissolved by any appropriate variation in the physical conditions. Generally, the pressure or temperature of the gaseous phase can be varied, after the gaseous phase is separated from the solid residue of the rubber. Depending on the circumstances, either an increase or diminution of pressure or temperature, or both pressure and temperature, may be used to condense the degradation products of the rubber. The degradation products may, particularly if either the pressure or temperature is diminished, contain some of the organic solvent but this can generally be separated from the degradation products by distillation.

The following is a non-limitative example illustrating the invention:—

A worn car tyre was chopped by hand into pieces weighing roughly 5 g. 0.800 kg of the chopped tyre were placed in an autoclave together with 4.858 kg of toluene. At 150°C, vapour was removed containing 0.230 kg of toluene and 0.007 kg of water. The heating was then continued to a pressure of 7620 kN/m<sup>2</sup> at 365°C. The reactor contents were stirred for 30 minutes and then allowed to settle for 15 minutes, while maintaining the temperature at 365°C. The vapour phase was then released from the reactor through a condenser while maintaining the reactor at 365°C until the contents of the reactor were at atmospheric pressure. A total of 4.955 kg of condensate was recovered, which was distilled to 0.589 kg of black tar. 0.381 kg of a free flowing

powder was left as the residue of the original rubber.

#### WHAT WE CLAIM IS:—

1. A method of degrading rubber and recovering useful material therefrom, comprising contacting the rubber with a solvent selected from water and organic solvents having a critical temperature of between 150°C and 450°C, which solvent is in the gaseous phase, at above the critical temperature thereof, thereby degrading the rubber and dissolving at least part of the degradation products thereof in the gaseous phase, removing the gaseous phase from the solid residue of the rubber and condensing at least part of the degradation products of the rubber dissolved in the gaseous phase from the gaseous phase.

2. A method as claimed in claim 1 in which the solvent is an organic solvent having a critical temperature of between 250°C and 400°C.

3. A method as claimed in claim 1 or claim 2 in which the rubber is contacted with the solvent at a temperature of not more than 100°C above the critical temperature of the solvent.

4. A method as claimed in any of the preceding claims in which the rubber is vulcanised rubber.

5. A method as claimed in claim 4 in which vehicle tyres are a source of the rubber.

6. A method as claimed in any one of the preceding claims in which the degradation products of the rubber are condensed from the gaseous phase by a diminution of either the pressure or the temperature of the gaseous phase.

7. A method of recovering useful material by degrading rubber substantially as described herein with reference to the example.

8. Degradation products of rubber, condensed from the gaseous phase, whenever produced by a method as claimed in any one of the preceding claims.

9. Residues from rubber products from which at least a portion of the degradation products of rubber have been removed into a gaseous phase by a method as claimed in any of claims 1 to 7.

For the Applicants,  
J. I. WOOD,  
Chartered Patent Agent.